BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.

(11) (A) No. 1 123 792

(45) ISSUED 820518

(52) CLASS 210-49

(51) INT. CL. ^{3} B65D 41/50

(19) (CA) **CANADIAN PATENT** (12)

(54) MERCURY SEALABLE PLUG

(72) Knapp, William C.;
Trizisky, Joseph D.,
Canada

(73) Granted to Du Pont Canada Inc.
Canada

(21) APPLICATION No. 282,025

(22) FILED 770705

No. OF CLAIMS 7

DISTRIBUTED BY THE PATENT OFFICE, OTTAWA.
CCA-274 (3-80)

ABSTRACT OF THE DISCLOSURE

A sealing device, i.e., a mercury sealable plug, for a neck of a vessel is disclosed. The sealing device comprises a body having a first end and a second end; the first end being adapted to face outwardly from the neck of the vessel and the second end being adapted to be within the vessel. The body has a longitudinal passageway running therethrough with outlets at the first and second ends of the body. Two annular grooves in the outer side surface of the body are adapted to contain two annular rings capable of providing a seal with the inner surface of the neck of the vessel. The outlets at the first and second ends of the body are adapted to be closed by first and second septums respectively. The passageway and an annular space defined by the two rings, the outer surface of the body and the inner surface of the neck are adapted to contain mercury to seal the vessel opening in such a way that any contents of the vessel may be sampled by means of a syringe needle through the first septum, the passageway and the second septum over an extended period of time without gases diffusing to or from the vessel between samplings. In an embodiment of the sealing device, at least one channel leads from the passageway to the outer side surface of the body between the two annular grooves. The channel is also adapted to contain mercury and to connect the passageway to the annular space.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A sealing device for a neck of a vessel comprising:

a body having a first end and a second end, the first end being adapted to face outwardly from the neck of the vessel and the second end being adapted to be within the vessel,

a passageway running longitudinally through the body and having an outlet at the first end and at the second end,

two spaced annular grooves in the outer side surface of the body, the grooves being adapted to contain two annular rings capable of providing a seal with the inner surface of the neck of the vessel,

the outlet at the first end of the body being adapted to be closed by a first septum,

the outlet at the second end of the body being adapted to be closed by a second septum, the passageway and an annular space defined by the two rings, the outer side surface of the body and the inner surface of the neck being adapted to contain mercury to seal the vessel neck in such a way that any contents of the vessel may be sampled by means of a syringe needle through the first septum, the passageway and the second septum over an extended period of time without gases diffusing to or from the vessel between samplings.

2. The sealing device of Claim 1 including at least one channel leading from the passageway to the outer

side surface of the body between the two annular grooves, the channel also being adapted to contain mercury and to connect the passageway to the annular space.

3. The sealing device of Claim 2 in which the two annular rings, the first septum and the second septum are made of an elastomeric material.

4. The sealing device of Claim 3 in which the body is made of stainless steel.

5. The sealing device of Claim 3 in which the second septum is recessed into the body and held in position by a threaded plug, a passage running axially through the threaded plug adapted to permit a syringe needle to be passed therethrough.

6. The sealing device of Claim 5 in which the first septum is held in position by an aluminum cap, an opening in the aluminum cap being adapted to permit a syringe needle to be passed therethrough.

7. The sealing device of any one of Claim 3, Claim 4 and Claim 6 in which the two annular rings, the first septum and the second septum are made of silicone rubber.

This invention relates to a sealing device for a neck of a vessel which permits access to the contents of vessel by means of a syringe needle while providing an effective seal against long-term gaseous diffusion to or from the vessel.

Sealing devices for a neck of a vessel which permit access to the contents of the vessel by means of a syringe needle are known in the art. However, such sealing devices generally consist of septums of one or more layers of elastomeric material. The elastomeric material provides an adequate seal against gaseous diffusion to or from a vessel for short periods e.g. less than a week. However, where the contents of the vessel are to be sampled by a syringe needle over long periods e.g. greater than one month, gaseous diffusion through the elastomeric material may become a problem. For example, in a study of the degradation of polyethylene in air at room temperature over a period of several months in a vessel closed by a sealing device comprising septums of elastomeric material, diffusion of oxygen (from air) through the elastomeric material into the vessel was sufficiently rapid to completely obscure any change in the oxygen content of the vessel as a result of degradation of the polyethylene.

It is an object of the present invention to provide a sealing device for a neck of a vessel which permits access to the contents of the vessel by means of a syringe needle while providing an effective seal against gaseous diffusion to or from the vessel over long periods of time.

It has now been found that such a sealing device may be provided if mercury is utilized between septums as

a sealing medium.

Accordingly, the present invention provides a sealing device for a neck of a vessel, comprising:

a body having a first end and a second end, the first end being adapted to face outwardly from the neck of the vessel and the second end being adapted to be within the vessel,

a passageway running longitudinally through the body and having an outlet at the first end and at the second end,

two spaced annular grooves in the outer side surface of the body, the grooves being adapted to contain two annular rings capable of providing a seal with the inner surface of the neck of the vessel,

the outlet at the first end of the body being adapted to be closed by a first septum,

the outlet at the second end of the body being adapted to be closed by a second septum, the passageway and an annular space defined by the two rings, the outer side surface of the body and the inner surface of the neck being adapted to contain mercury to seal the vessel neck in such a way that any contents of the vessel may be sampled by means of a syringe needle through the first septum, the passageway and the second septum over an extended period of time without gases diffusing to or from the vessel between samplings.

In an embodiment of the present invention, the sealing device includes at least one channel leading from the passageway to the outer side surface of the body between the two annular grooves, the channel also being adapted to contain mercury and to connect the passageway to the annular space.

In another embodiment of the present invention, the two annular rings, the first septum and the second septum are made of an elastomeric material.

In yet another embodiment, the body of the sealing device is made of stainless steel.

In a further embodiment the two annular rings, the first septum and the second septum are made of silicone rubber.

An embodiment of the invention is illustrated by reference to the accompanying drawings in which:

Fig. 1 is a half section view of a sealing device according to one embodiment of the present invention shown positioned in a neck of a vessel (not shown).

Fig. 2 is a section view of the embodiment of Fig. 1 viewed in the direction of arrows II-II in Fig. 1.

Fig. 3 is a plot of the ratio of the areas of peaks on a gas chromatograph for oxygen and nitrogen with time, in hours, for gas samples sealed in sample tubes using the sealing device of the present invention, and for comparison using a sealing device that did not contain mercury.

In Figures 1 and 2, a sealing device according to one embodiment of the present invention is designated generally by the numeral 10 and is shown mounted in a neck 11 of a vessel (not shown). The sealing device 10 has a body 12. Body 12 has a first end facing outwardly from neck 11 of the vessel and a second end within the vessel. A passageway 13 running longitudinally through body 12 has an outlet 14 at the first end and an outlet 15 at the second end. Two annular grooves 16 in the outer side surface of body 12 each contain an annular ring 17 which seals against the inner surface of the neck 11. Six channels 18 (see Fig. 2) lead from the

passageway 13 to the outer surface of the body 12 between the two annular grooves 16. A first septum 19, held in position by an aluminum cap 20 having an opening 21 therein covers the outlet 14 in the first end of body 12. It will be appreciated that the aluminum cap 20 may be omitted or that other means to hold the first septum 19 in position may be substituted therefor. A second septum 22, which is recessed into the body 12 and held in position by a threaded plug 23, covers the outlet 15 at the second end of body 12. A passage 24 runs axially through threaded plug 23. It will be appreciated that means other than threaded plug 23 may be used to hold the second septum 22 in position. The passageway 13 (between first septum 19 and second septum 22), the channels 18 and the annular space defined by the two annular rings 17, the outer surface of body 12 and the inner surface of the neck 11 are full of mercury.

In operation, the sealing device 10, with the first septum 19 and the aluminum cap 20 removed, is positioned in the neck 11 of a vessel, the contents of which are to be sampled repeatedly over an extended period of time by means of a syringe needle. Mercury is poured into the passageway 13, from which it flows through the six channels 18 into the annular space defined by the two annular rings 17, the outer side surface of body 12 and the inner surface of the neck 11 of the vessel. It will be appreciated that the number of channels 18 is not critical, for example, one channel may be sufficient to allow the mercury to flow from the passageway 13 into the annular space. Under some conditions, for example, where sampling is being carried out very frequently over an extended period of time it may be desirable to eliminate channels 18 entirely. In these circumstances mercury is added to the annular space by means of a syringe needle which is passed

through the upper annular ring 17. A syringe needle is also used to remove air trapped between the mercury and the upper annular ring 17 during the addition of the mercury. When the passageway 13 is full of mercury, the first septum 19 is positioned over the outlet 14 of channel 13 and held in position by the aluminum cap 20.

In sampling the vessel contents, a syringe needle is passed in series through: the opening 21 in the aluminum cap 20; the first septum 19; the passageway 13; the second septum 22; and the passage 24 of the threaded plug 23; into the vessel. The presence of the mercury in the passageway 13 and in the annular space defined by the two annular rings 17, the outer side surface of body 12 and the inner surface of the neck 11 of the vessel, makes it possible to sample the vessel contents repeatedly over extended periods of time, e.g. several months, without any gases diffusing to or from the vessel between samplings.

In instances where, during the sampling period, the neck 11 of the vessel may be positioned for any extended period of time in a near horizontal position, it is important that the passageway 13 remain full of mercury (to ensure that the annular space between annular rings 17 remains sealed with mercury). On each sampling of the vessel contents, a small quantity of mercury may escape through the first septum 19 with the syringe needle as it is withdrawn therefrom. Thus, where sampling is to be carried out very frequently over an extended period, it may be desirable to add mercury to passageway 13 occasionally during the sampling period. The mercury may be added through first septum 19 by means of a syringe needle. Alternatively, the problem of ensuring that the annular space between annular rings 17 remains sealed

with mercury may be solved by the elimination of channels 18 as described heretofore.

The body 12 may be made from any suitable material, for example metal, which is inert to mercury and to the vessel contents and is impermeable to gases. A preferred material for the body 12 is stainless steel.

The two annular rings 17, the first septum 19 and the second septum 22 may be made from any suitable material which will allow passage of a syringe needle therethrough and which is resilient anough to keep to a minimum any leakage of mercury which may occur when the syringe needle is withdrawn therefrom. Preferred materials are elastomeric materials and a more preferred material is silicone rubber.

The present invention is illustrated by the following example.

EXAMPLE

Two sealing devices according to the present invention as illustrated in Figures 1 and 2 were mounted in separate sample tubes, one containing a gas mixture of 15% oxygen, by weight, in nitrogen and the other containing a gas mixture of 24% oxygen, by weight, in nitrogen. Two other sealing devices identical to the one illustrated in Figures 1 and 2 except that the mercury has been omitted from the sealing devices were mounted in two other sample tubes, one containing a gas mixture of 15% oxygen, by weight, in nitrogen and the other containing a gas mixture of 24% oxygen, by weight, in nitrogen.

In each of the above four sealing devices, the body 12 and the plug 23 were made of stainless steel and the two annular rings 17, the first septum 19 and the second septum 22 were made of silicone rubber.

The four sample tubes were stored for an extended period of time. Samples of the gas mixture in each sample tube were taken periodically with a syringe needle, as hereinabove described, and analyzed using a gas chromatograph.

In Figure 3, the ratio of the areas of peaks on a gas chromatograph for oxygen and nitrogen for the gas mixture in each sample tube is plotted versus time in hours. The ratio of the areas under the respective peaks on the gas chromatograph is indicative of the amount of oxygen present in the tubes. In the absence of diffusion of oxygen and/or nitrogen through the sealing devices, it would be anticipated that the ratio of the peak areas for oxygen : nitrogen would remain constant throughout the sampling period. The curves in Figure 3 may be identified as follows:

(1) the closed squares - indicate samples from the sample tube initially containing 15% by weight of oxygen and closed by the sealing device of the present invention;

(2) the closed circles - indicate samples from the sample tube initially containing 24% by weight of oxygen and closed by the sealing device of the present invention;

(3) the open squares - indicate samples from the sample tube initially containing 15% by weight of oxygen and closed by a sealing device from which mercury had been omitted;

(4) the open circles - indicate samples from the sample tube initially containing 24% by weight of oxygen and closed by a sealing device from which mercury had been omitted; and

(5) the horizontal dotted line - indicates the arithmetic means obtained for measurements of the oxygen : nitrogen ratios of a number of air samples using the gas chromatograph.

From Figure 3 it is apparent that the gas compositions in the sample tubes sealed with the sealing devices without mercury approach the composition of air after a period of about 2400 hours due to gas diffusion through the silicone rubber annular rings 17 and/or through the silicone rubber first and second septums 19 and 22, respectively. It is also apparent from Figure 3, that the gas compositions in the sample tubes sealed with the sealing device of the present invention show no apparent change after a period of about 1800 to 2000 hours.

2-1

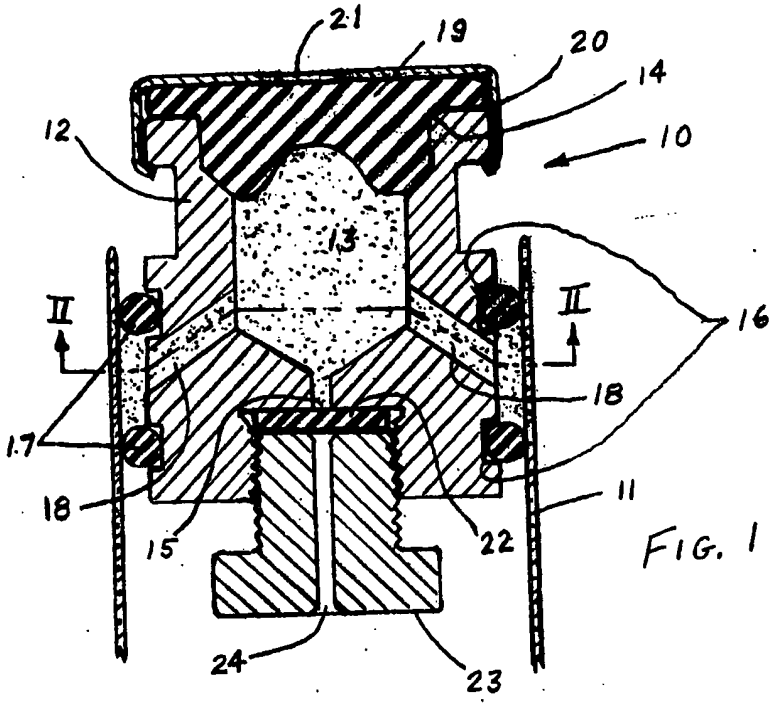


FIG. 1

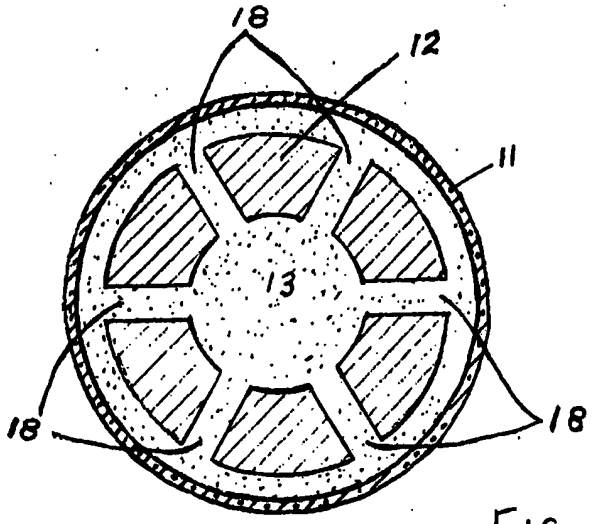
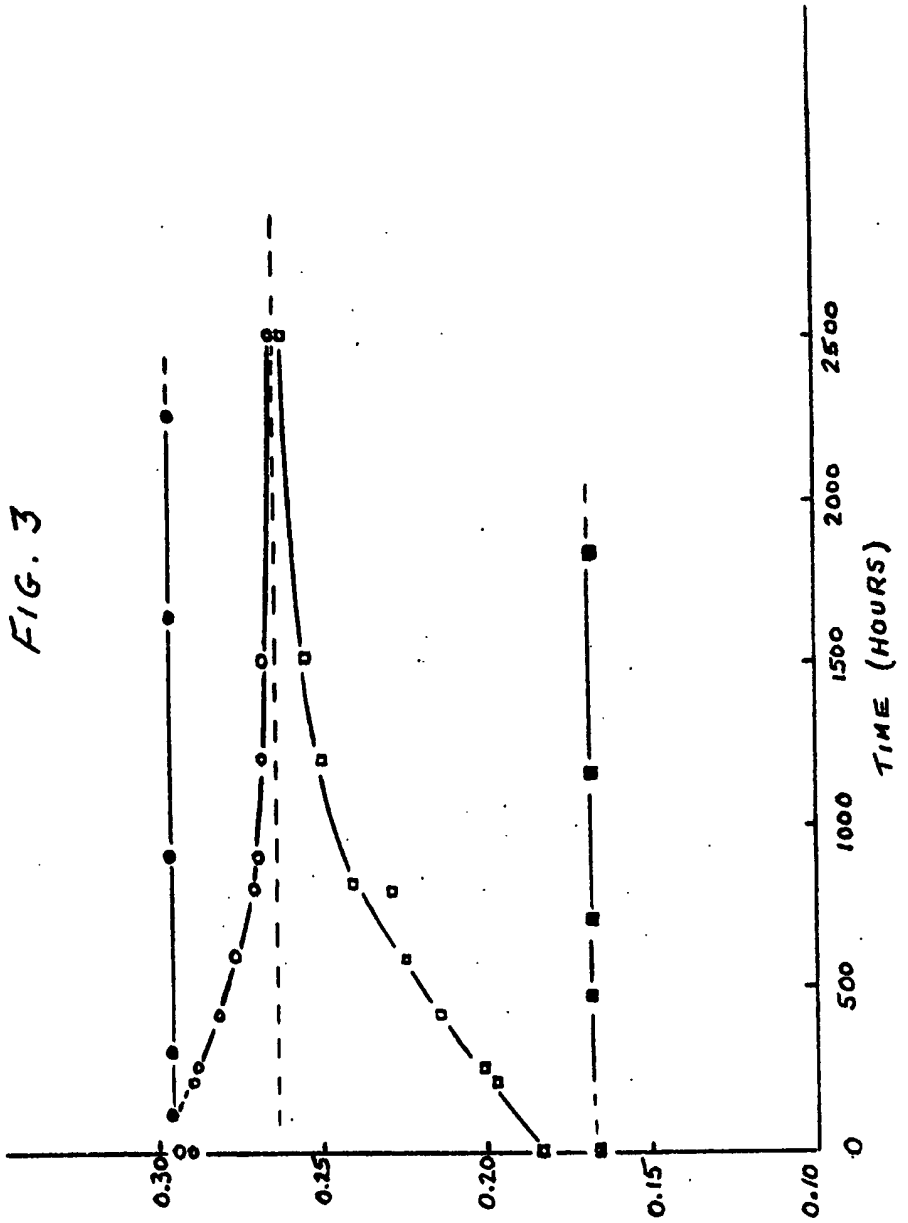


FIG. 2

WILLIAM C. KNAPP
&
JOSEPH D. TRIZISKY

McCallum, Brooks & Co.
PATENT AGENTS

FIG. 3



RATIO OF THE AREAS OF PEAKS ON A GAS CHROMATOGRAPH FOR OXYGEN AND NITROGEN

WILLIAM C. KNAPP
JOSEPH D. TRIZISKY
McCallum, Burks & Co.
PATENT AGENTS